

THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

HAYASHI, Hideo et al.

Group Art Unit: 1623

Application No. 10/510,952

Examiner: Michael C. Henry

Filed: 03/16/2005

Confirmation No.: 8837

For: METHOD FOR MODIFYING GUM

ARABIC

Commissioner for Patents PO Box 1450 Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 C.F.R. § 1.132

- I, Tsuyoshi KATAYAMA , do hereby make the following declaration:
- I am a Japanese citizen, residing at 1-1-11,
 Sanwa-cho, Toyonaka, Osaka, 561-8588, Japan.
- 2. I graduated from Okayama University, Faculty of Agriculture, Department of agricultural chemistry in 1992. I also graduated from the Graduate School of Natural Science and Technology, and received a Master's Degree in 1994.
- 3. I also graduated from the Graduate School of Natural Science and Technology, and received a phD Degree in 2006.
- 4. I began my employment with SAN-EIGEN F. F. I., INC., the assignee of the above-identified application, on April in 1994. Since 1994, I have been engaged in the research and development of Emulsion and it's emulsifier. I am working for the colloids science laboratory since 2006, and I am presently engaged in research and development of gum arabic.

5. I am one of the named inventors of the above-identified application, and am familiar with the subject matter of said application as well as the disclosures in the cited references.

In order to compare the present invention and the prior art teachings, I have conducted the following experiments.

EXPERIMENT

I. Purpose

The purpose of this experiment is to evaluate the change in appearance, emulsifying ability, and viscosity of the following three samples, after allowing the samples to stand under the following conditions at a predetermined relative humidity and heating temperature.

II. Materials and Method

II-1. Materials

- Test Sample 1

Coarsely ground gum arabic (average particle diameter: 1 mm)

- Test Sample 2

Coarsely ground gum arabic (average particle diameter: 5 mm): the same gum arabic used in the Examples in the present specification

- Comparative Sample 1

Gum arabic mechanical powder (average particle diameter: 300 μ m): presumably the same gum arabic used in the test by Wilson et al.

II-2. Method

The change in appearance and emulsifying ability were evaluated by the method described in the Examples in the present specification. The viscosity was measured as the rotational viscosity of 10% solutions (Brookfield viscosity at 20°C, rotor

No. 1). Specifically, the evaluation and measurement were carried out as follows.

One (1) kilogram portions of gum arabic (Test Samples 1 and 2, and Comparative Sample 1) were separately placed and allowed to stand in hermetically sealed constant-humidity vessels adjusted to a relative humidity of 85% and a temperature of 88°C (i.e., 190°F) for 12 hours, and then cooled to room temperature (25°C) in the same humidity environment to give modified gum arabic samples. These conditions are substantially the same as those in the Preparation Examples 26 and 27 in the present specification.

The changes in appearance of the modified gum arabic samples obtained by the above method were investigated by the method described hereinafter. Furthermore, each of these modified gum arabic samples was used to prepare an emulsion, and the average particle diameter was investigated and the emulsifying ability of each emulsion was evaluated. In addition, the viscosity of the modified gum arabic samples obtained by the above method was measured.

(1) Change in Appearance

The appearance of the modified gum arabic samples obtained as above was compared by the naked eye with the unmodified gum arabic, and observed for agglomeration of particles (mass formation) and dehydration.

(2) Evaluation of Emulsifying Ability

One (1) kilogram portions of the modified gum arabic were separately dissolved in 4 kg of water, centrifuged to remove insolubles, and made into 20 weight % aqueous gum arabic solutions. To each of these 20 weight % aqueous gum arabic solutions(850g) was added 150 g of medium-chain triglyceride (octanoic/decanoic acid triglyceride O.D.O[™] (product of the Nisshin Oil Mills)) under agitation, and the mixture was

emulsified using a homogenizer (manufactured by APV Gaulin) (homogenized 4 times at a pressure of 4.4 MPa (450 kg/cm 2)). The average particle diameter of the resulting emulsion was measured immediately after emulsification and after 7 days of storage at 60°C using a particle size distribution analyzer SALD-1100 (a laser diffraction system, manufactured by Shimadzu Corporation).

The emulsifying ability of an emulsifier is generally evaluated as follows. The smaller the average particle diameter of the emulsion obtained is and the longer the particle diameter of the emulsion is sustained stably, the higher the emulsifying ability is.

(3) Measurement of Viscosity

The rotational viscosity of 10% solutions of the samples was measured (Brookfield viscosity at 20°C, rotor No. 1).

III. Results

The results of studies on the above (1)-(3) are presented in Table 1.

Table 1

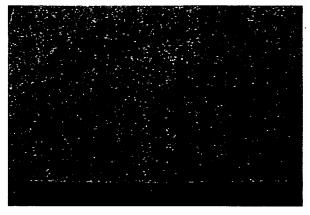
| | | | Emul | Emulsifying ability | ity | |
|---------------------|--------------|-----------|-------------|---------------------|------------|------------------------------|
| | | | (medi | (median diameter: | hm) | |
| Sample of cames | Treatment | Viscosity | Immediately | After | Difference | Evaluation of Appearance |
| טיר לייישר מיישר | דבמרווופזזר | (CP) | after | storage at | (b-a) | הימיוטיוט כו האף כמומוני |
| | | | preparation | 60°C for 7 | | |
| | | | (a) | days (b) | | |
| Comparative | Before | | | | | |
| Sample 1: | treatment | 11 | 1.35 | 9.25 | 7.80 | - (Photograph 1) |
| Gum arabic | (unmodified) | | | | | |
| mechanical powder | | | - | | | Heavily caked and completely |
| (average particle | After | | | | | solidified into a syrupy |
| diameter: 300 µm) | treatment | 18. | * | * | * | mass; could not be removed |
| | (modified) | | | | | from the container |
| | | | | | | (Photograph 2). |
| Test Sample 1: | Before | | | | | |
| Coarsely ground | treatment | 11 | 1.25 | 9.00 | 7.75 | - (Photograph 3) |
| gum arabic | (unmodified) | | | | | |
| (average particle | | | | | | Slightly caked with partial |
| diameter: 1 mm) | | | | | | agglomeration of particles, |
| | Before | | | | | but not solidified into a |
| | treatment | 20 | 0.68 | 0.73 | 0.05 | syrupy mass; could be |
| | (modified) | | | | | removed from the container |
| | | | | | | with no practical problems |
| | - | | | | | (Photograph 4) |
| Test Sample 2: | Before | | | | | |
| Coarsely ground | treatment | 11 | 1.20 | 8.85 | 7.65 | - (Photograph 5) |
| gum arabic | (unmodified) | | | | | |
| (average particle | | | | | | Partial agglomeration of |
| diameter: 5 mm) | After | | | | | ground particles, but |
| | treatment | 22 | 0.65 | 0.68 | 0.03 | without any marked |
| | (modified) | | | | | abnormalities (Photograph |
| | | | | | | 6) |

* Comparative Sample 1 (gum arabic mechanical powder with an average particle diameter of 300 µm) solidified into a syrupy mass after treatment. The solidified sample could not be dissolved to evaluate the emulsifying ability.

Photograph 1

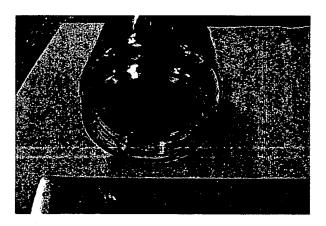
Comparative Sample 1

Unmodified gum arabic powder (average particle diameter: 300 μm)



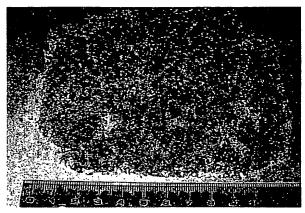
Photograph 2

Modified gum arabic powder (average particle diameter: 300 $\mu m)$ Heated at 88°C and a relative humidity of 85% for 12 hours



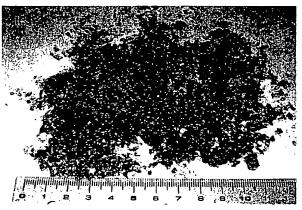
Photograph 3

Unmodified coarsely ground gum arabic (average particle diameter: 1 mm)



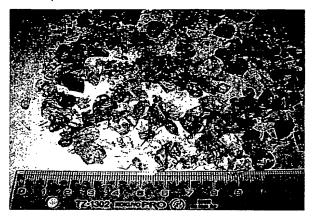
Photograph 4

Coarsely ground gum arabic (average particle diameter: 1 mm) Heated at 88°C and a relative humidity of 85% for 12 hours



Photograph 5

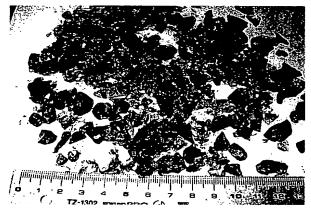
Unmodified coarsely ground gum arabic (average particle diameter: 5 mm)



Photograph 6

Modified coarsely ground gum arabic (average particle diameter: 5 mm)

Heated at 88°C and a relative humidity of 85% for 12 hours



IV. Discussion

As is apparent from the above results, modified gum arabic with good appearance, emulsifying ability and viscosity was obtained when gum arabic with an average particle diameter of 1 mm or 5 mm was used as the starting material.

In contrast, when gum arabic with an average particle diameter of $300~\mu m$ was used, the gum arabic underwent a marked color change, agglomerated into a syrupy mass, and adhered to the container. Thus,

when gum arabic with an average particle diameter of 300 μm was used, good modified gum arabic could not be obtained.

I, the undersigned, declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

| Date: | 16.7.2008 | | |
|-------|-----------|----|-------------------|
| | | ву | Jsvyoshi Katazama |